

**National Exposure Research Laboratory  
Exposure Methods and Measurement Division**

**Quality Assurance Project Plan  
Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Substances  
(PFAS) in Environmental Media from an Industrialized Area of New Jersey**

Safe and Sustainable Water Resources / Project ID / 6.02

**September 14, 2017**

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Note: This QAPP was conditionally approved upon receipt on September 14, 2017. Signatures were collected after clarifications to the QAPP were completed.

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Mark J. Strynar <a href="mailto:strynar.mark@epa.gov">strynar.mark@epa.gov</a> 919-541-3706	ORD/NERL/EMMD/PHCB	PI & Technical lead/analytical chemistry	Lead analyst for water, targeted and non-targeted, data analysis, manuscript preparation
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Adam Biales <a href="mailto:biales.adam@epa.gov">biales.adam@epa.gov</a> 513-569-7094	ORD/NERL/EMMD/IEIB	IEIB Chief	Review and approve the project QAPP
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Nidal Azzam azzam.nidal@epa.gov 212-637-3748	USEPA Region 2	Region 2 coordinator/ liaison	Manage project and communications within Region 2, manuscript preparation
Erica Bergman erica.bergman@dep.nj.gov 609-292-7406	New Jersey Department of Environmental Protection (NJ DEP)	NJ DEP project manager	Manage overall project coordination, communications within NJ DEP, data analysis, manuscript preparation
Sandra Goodrow sandra.goodrow@dep.nj.gov 609-984-9556	New Jersey Department of Environmental Protection (NJ DEP)	NJ DEP project coordinator	Manage field sampling efforts, data analysis, manuscript preparation

## A5. Problem Definition and Background

The Environmental Protection Agency's (EPA) National Exposure Research Laboratory (NERL), the EPA Region 2, and the New Jersey Department of Environmental Protection (NJ DEP) plan to conduct a joint research effort designed to characterize poly- and perfluoroalkyl substance (PFAS) contamination in southwestern New Jersey. Multiple public water systems and private wells in this area have been found with PFAS contamination. However, the sources, environmental transport mechanisms, and the geographical extent of this contamination are not well known. This collaborative research effort will help NJ DEP with its ongoing efforts to identify and remediate PFAS-contaminated drinking water resources, explore the full extent of contamination in this region, and identify the populations at risk to PFAS exposures.

Discharges to surface water by major industrial sources and potentially other smaller facilities have contaminated the Delaware River, its tidal estuaries, and many underlying aquifers, and this has led to the ongoing presence of PFAS in drinking water resources in this area (DRBC, 2012, Post et al., 2013). Previous analysis of surface water and groundwater suggests specific PFAS source signatures and the potential to determine their specific origins by comparing ratios of various PFAS in different samples (Post et al., 2013). PFAS were also known to be present in airborne emissions from local industrial sources (Integral Consulting, 2015), but the potential impacts from this route of distribution remain uncharacterized. An additional complication is that regulatory pressure has caused industries to shift to a new class of PFAS in recent years (Wang et al., 2013). These new PFAS have not been identified or quantitated, and the potential for their environmental presence and related human exposures remains unknown.

The overall objectives of this project therefore include a confirmation of ongoing surface and groundwater contamination from current and historical source discharges, establishing specific PFAS source signatures originating from different industrial sites, and an evaluation of the potential for regional air deposition impacts. Identification of any new PFAS may also be possible using advanced analytical techniques.

To help accomplish these objectives we specifically plan to collect and analyze up to 57 water samples from tidal and non-tidal surface water bodies and ground water from private drinking water wells, up to 48 soil/sediment samples, and up to 48 plant samples as described below.

## **A6. Project/Task Description**

### **Study Area**

The information describing the sample study area is covered in the previously prepared study plan entitled “Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Substances (PFAS) in Environmental Media from an Industrialized Area of New Jersey” revised August 23, 2017. Please refer to the study plan document to access all Figures and Tables described in the following “Sampling Approach” section.

Under the direction of the NJ DEP Project Manager, the NJ DEP will collect samples in three southwestern New Jersey counties in locations along or near the Delaware River in areas previously known to have been impacted by PFAS and at one location that extends into a fourth county (Burlington) (see Figures 1-5 of the study plan). NJ DEP will also collect samples from two reference sites farther north, to establish background levels of target analytes.

### **Sampling Approach**

#### *Surface water discharges*

To confirm previous monitoring results and evaluate the ongoing impacts from historic and current surface water discharges, 20 water samples will be collected by NJ DEP from local tidal waterways (Figure 1 and Table 1) and 25 private wells (Figure 2 and Table 2) in the study area. Sediment samples will also be collected from each surface water collection site (Figure 1 and Table 1) to help further characterize contamination of these water bodies.

#### *Atmospheric Transport*

Previous research has documented that airborne emissions from industrial sources can lead to long range atmospheric transport of PFAS and contamination of soils, surface water, and groundwater in areas distant from the emission point (Davis et al., 2007). This transport mechanism has not been evaluated in the New Jersey study area yet but it is important to consider because it could greatly increase the size of the impacted area and the number of people who may be exposed.

To investigate this potential transport mechanism, 24 surface soil samples plus 4 soil cores will be collected by NJ DEP to help characterize historical deposition patterns (Figures 3 and 4, Tables 3 and 4). The soil samples will be collected along transects following the main prevailing wind directions downwind of known sources (Figure 3, Table 3, and Figure 5). Two of these samples will be collected from background sites thought not to be potentially impacted by the airborne sources (Sites 21 and 22 on Figure 3 and in Table 3). At most soil-sampling locations, a plant sample will be collected to investigate plant uptake from the investigated soils.

To establish further evidence of possible impacts from airborne emissions, 11 additional surface water samples will be collected by NJ DEP from ponds downwind of the sources but hydrologically isolated from potential inputs from the Delaware River and its tidal flows (Figure 5 and Table 5). Many of the soil sample locations listed in Table 3 are either adjacent or in close proximity to these ponds, making it possible to evaluate whether runoff from or percolation through contaminated soils is related to the PFAS measured in these isolated water bodies. In addition, two soil sample locations (Sites 1 and 2 in Figures 4 and 5) are in close proximity to both pond sites and drinking water samples located near tidal tributaries, allowing for comparison of PFAS patterns measured at each location and evaluation of predominant transport mechanisms.

## **Methods**

### *Field Collection*

Field collection will be led by teams at NJ DEP in cooperation with consulting firms currently collecting samples in the area for the State. NJ DEP will also cooperate with the Delaware River Basin Commission (DRBC) where sediment and surface water samples will be taken from locations familiar to DRBC. Field collection SOPs for all media are identified as the following:

New Jersey Department of Environmental Protection, Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Compounds in Environmental Media from an Industrialized Area of New Jersey: Work Plan for Groundwater (Private Well) Sampling, 29 August 2017.

New Jersey Department of Environmental Protection, Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Compounds in Environmental Media from an Industrialized Area of New Jersey: Work Plan for Tidal Surface Water and Sediment Sampling, 31 August 2017.

New Jersey Department of Environmental Protection, Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Compounds in Environmental Media from an Industrialized Area of New Jersey: Work Plan for Soil and Non-Tidal Surface Water Sampling, 29 August 2017.

### *Analytical Support*

The analysis of all media collected will be performed by the Environmental Protection Agency's (EPA) National Exposure Research Laboratory (NERL) in Research Triangle Park, North Carolina and Athens, Georgia. Surface water and well water samples will be analyzed for PFAS using the three SOPs:

Improved Method for Extraction and Analysis of Perfluorinated Compounds (PFCs) from Surface Waters and Well Water by Ultra-High Performance Liquid Chromatography (UPLC)-Tandem Mass Spectrometry (MS/MS) (D-EMMD-PHCB-043-SOP-03).

Method for Extraction and Analysis of Perfluoroethercarboxylic acids (PFECAs) from Surface Water, Well Water and Waste Water by Ultra-High Performance Liquid Chromatography (UPLC)-Tandem Mass Spectrometry (MS/MS) (D-EMMD-PHCB-062-SOP-01).

Analytical method for non-targeted and suspect screening in environmental and biological samples using Time of Flight Mass Spectrometry (TOFMS) (D-EMMD-PHCB-034-SOP-01).

Soil and sediment samples will be analyzed using methods described in Rankin et al. (2016) and the draft NERL SOP Exhaustive Extraction of Charged Per- and Polyfluorinated Alkyl Substances (PFASs) from Contaminated Soil & Sediment Samples with Ion-Pairing Cleanup (D-EMMD-ECB-010-SOP-01).

Plant sample analyses are planned to be performed by adapting the above soil methods or following Yoo et al. (2011).

The core analytes for all matrices include perfluorobutanoic acid (C4), perfluoropentanoic acid (C5), perfluorohexanoic acid (C6), perfluoroheptanoic acid (C7), perfluorooctanoic acid (C8 or PFOA), perfluorononanoic acid (C9 or PFNA), perfluorodecanoic acid (C10), perfluorobutane sulfonate (PFBS), perfluorohexane sulfonate (PFHS) and perfluorooctane sulfonate (PFOS) (except for soil, which does not include C4 and C5). The occurrence of additional precursor and "replacement" PFAS will be evaluated using the non-targeted and suspect screening high resolution mass spectrometry.

After QA review, analytical results will be conveyed to NJ DEP in the form of an Excel spreadsheet containing raw analytical instrument outputs (e.g., retention times, area counts, area ratios), all details relating to calibration curves, and final analyte concentrations with annotations as needed to flag or describe notable results.



[illegible]

The overall QA objective of the study is to obtain 100% true data. This ideally is a combination of 100% retention from field collection, shipping, and storage, 100% recovery through laboratory preparation and purification processes, and finally 100% accuracy and precision with all analytical methods used. Since we recognize that it not practically possible to reach this goal (because of human error, instrument variability, and/or suboptimal performance of methods) a detailed list of all performance objectives follows below:

$$\text{Relative \% Difference} = [(\text{conc.1} - \text{conc. 2}) / (\text{mean of conc.1 and 2})] \times 100\%$$

**Accuracy:** Accuracy is reported as percent difference between the concentration of a sample prepared by spiking a target compound into a blank matrix and concentration being determined using a standard curve which is compared to the theoretical calculated concentration recorded in

the laboratory notebook. Target accuracy is  $\pm 30\%$  of the nominal theoretical calculated value or 30% of the calculated value at the lowest point on the calibration curve.

Accuracy = (spike level determined by standard curve / theoretical spike concentration) x 100%

Representativeness: Samples collected in this effort will be obtained from locations determined to be suitable by the NJ DEP. We acknowledge that the resulting data will not be representative of all small details in this region but they should be sufficient to meet the objectives stated above and possibly represent larger-scale patterns.

Completeness: We anticipate >95% completeness for water, soil, and sediment samples that are shipped to NERL for analysis. Losses may occur due to loss, leakage of collection bottles, or spills during the analytical procedures.

Comparability: All of the procedures used in this assessment will be based on methods that have been used for previously published research conducted in our laboratories. Authentic standards obtained from commercial sources will be used to assure accurate compound identification and reliable quantitation when possible. Analysis of standard reference materials (SRM) will also be included in the analyses when possible.

Method sensitivity: The lower limit of quantitation (LLOQ) is defined as the lowest point of calibration curve at which level the signal-to-noise ratio was at least 10:1 with precision of 15% (CV) and accuracy of 100% $\pm$ 30%.

We note that the USEPA's Office of Water (OW) has established a drinking water Health Advisory for the sum of PFOS and PFOA at 70 ng/L. Also, NJ Drinking Water Quality Institute (DWQI) has recommended health-based maximum contaminant levels (MCL) for drinking water of 14 ng/L for PFOA and 13 ng/L for PFNA. Given the stringency of these threshold levels the water analysis method used in this assessment will be optimized to give accuracy and precision in the 10 - 35 ng/L range. To that end, the field spikes used in this study will be prepared at 15 ng/L for all of the target analytes for which standards are available. Calibration curves will also be prepared with the primary intention of determining how the unknown water samples relate to this threshold.

#### **A8. Special Training/Certifications**

No specific training is required as for this project, but the analysts shall have completed all site specific health and safety training requirements that are applicable and be competent in the operations of the analytical instrumentation being used. Records of this training are maintained by the SHEM office or by individual researchers, respectively. This document assumes laboratory personnel will have a thorough working knowledge of basic laboratory skills, reagents, and instrumentation. The SOPs used are designed to guide a competent laboratory worker in the analysis of PFAS compounds, and they are not intended to instruct individuals on the basic aspects of analytical chemistry.

## A9. Documents and Records

### A9.1 Planning Documents

Three specific planning documents have been created to guide the development of this project. The first is the study plan entitled “Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Substances (PFAS) in Environmental Media from an Industrialized Area of New Jersey” dated August 23, 2017. As noted above, this document contains detailed maps and tables identifying all the sampling locations for this effort. The second is entitled “EPA Communication Plan PFAS Research Assistance for State of New Jersey” dated February 14, 2017. The third planning document prepared for this effort is this QAPP which outlines all of the essential elements needed for this study.

### A9.2 Tracking Documents

Drs. Mark Strynar and John Washington will retain copies of the chain of custody (CoC) records describing the deployment of field QC samples from NJ DEP.

Chain of Custody (CoC) forms will accompany the samples shipped to the EPA laboratory by NJ DEP, and these forms will be retained by EPA after analysis. This information will be maintained in a dedicated bound EPA laboratory notebook specific for this project and associated activities. Each analyst will maintain sample preparation records in laboratory notebooks, and LC/MS/MS analysis logs. The study file will also contain the records of QA issues, amendments to plans and SOPs, and corrective action reports. QA records, such as audit reports, corrective action reports, etc., will be maintained by the EPA PIs. Finally, Mark Strynar and John Washington will maintain the study file documentation including the final Excel spreadsheet of results, together with all associated final reports. All records will be maintained as per EPA specifications.

The Laboratory Research Notebooks (LRBs) is the place where records of the extraction and preparation of samples for analysis, the preparation of sampling containers, and the preparation of standard solutions for spiking and calibration are documented. Methods and reagents used are recorded and appropriate SOPs used for analysis are also cited.

The Sample Analysis records are the instrument logs that document the analyses of samples, and contain records of specific instrument conditions, and date and time of sample data acquisition.

## B. DATA GENERATION AND ACQUISITION

### B1. Experimental Design

Please refer to the study plan entitled “Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Substances (PFAS) in Environmental Media from an Industrialized Area of New Jersey” revised August 23, 2017 and details covered in Section A6 of this document.

## B2. Sampling Methods

Samples will be collected by NJ DEP, their contractors, and /or the Delaware River Basin Commission (described in section A6), using the procedures outlined in the following SOPs:

New Jersey Department of Environmental Protection, Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Compounds in Environmental Media from an Industrialized Area of New Jersey: Work Plan for Groundwater (Private Well) Sampling, 29 August 2017.

New Jersey Department of Environmental Protection, Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Compounds in Environmental Media from an Industrialized Area of New Jersey: Work Plan for Tidal Surface Water and Sediment Sampling, 31 August 2017.

New Jersey Department of Environmental Protection, Detection, Evaluation, and Assignment of Multiple Poly- and Perfluoroalkyl Compounds in Environmental Media from an Industrialized Area of New Jersey: Work Plan for Soil and Non-Tidal Surface Water Sampling, 29 August 2017.

## B3. Sample Handling and Chain of Custody

Chain of Custody forms will be generated by NJ DEP or their contractors using the following conventions:

Acronyms and sample naming conventions include using PF for PFAS Study, NSW for non-tidal surface water, SS for shallow soil, SC for soil core, and xxx for three-digit sample number (i.e., PFNSW001 thru PFNSW014, PFSC001 thru PFSC028, and PFSO001 thru PFSO0028). The acronyms BB, DUP, SB, or FB, respectively, shall be added to the end of the corresponding sample ID to identify the bottle blanks (e.g., PFNSW001BB), field duplicate samples (e.g., PFNSW001DUP), spiked blanks (e.g., PFNSW014SB), and field blanks (e.g., PFSO001FB).

All water samples will be collected in one liter HDPE containers, stabilized with the addition of 5 mL of concentrated nitric acid, and stored and shipped at ambient temperature to the EPA/RTP analytical laboratory. Samples treated in this manner will remain stable for at least 70 days.

All soil samples will be collected in one liter HDPE containers and stored and shipped on ice to the EPA/Athens analytical laboratory. Samples treated in this manner will remain stable for at least 30 days.

All sediment samples will be collected in one liter HDPE containers and stored and shipped on ice to the EPA/Athens analytical laboratory. Samples treated in this manner will remain stable for at least 30 days.

Chain of custody forms will be shipped with samples and include information such as collected by name/date, shipped by name/date, and received by name/date plus any additional sampling information necessary to document sampling location and special comments (e.g., sample leak).

#### **B4. Analytical Methods**

Water samples will be analyzed using three different SOPs each focused on a different range of PFAS:

Improved Method for Extraction and Analysis of Perfluorinated Compounds (PFCs) from Surface Waters and Well Water by Ultra-High Performance Liquid Chromatography (UPLC)-Tandem Mass Spectrometry (MS/MS) (D-EMMD-PHCB-043-SOP-03).

Method for Extraction and Analysis of Perfluoroethercarboxylic acids (PFECAs) from Surface Water, Well Water and Waste Water by Ultra-High Performance Liquid Chromatography (UPLC)-Tandem Mass Spectrometry (MS/MS) (D-EMMD-PHCB-062-SOP-01).

Analytical method for non-targeted and suspect screening in environmental and biological samples using Time of Flight Mass Spectrometry (TOFMS) (D-EMMD-PHCB-034-SOP-01).

Soil and sediment samples will be analyzed using methods described in Rankin et al. (2016) and the draft NERL SOP Exhaustive Extraction of Charged Per- and Polyfluorinated Alkyl Substances (PFASs) from Contaminated Soil & Sediment Samples with Ion-Pairing Cleanup (D-EMMD-ECB-010-SOP-01).

Plant samples will be analyzed using an adaption of the soil methods or by the method described in Yoo et al. (2011).

#### **B5. Quality Control**

Quality control criteria for water, soil, and sediment samples are discussed in detail in the SOPs referenced in this document. Please refer to these specific procedures for a complete description.

#### **B6/B7. Instrument/Equipment Calibration, Testing, Inspection, Maintenance**

Preventative maintenance will be performed on all instruments and equipment used according to the schedule defined in the appropriate SOP. Balances are inspected and maintained for cleanliness and mass calibration during each use. Balances and pipettes are calibrated annually by an accredited calibration service. A balance log is maintained and a reference weigh is verified prior to use.

The LC/MS/MS system is tuned by the manufacturer at least two times a year during regularly scheduled preventive maintenance service, or more frequently if conditions warrant. The system is manually tuned by the service technician to insure ion intensities, relative ion abundances, mass resolution, and ion peak shape are within manufacturer's specifications. Data on tune

performance parameters is available in the instrument log file. The calibration of the LC/MS/MS response for individual compounds is based on the calibration curve solutions that are analyzed with each batch that is analyzed. In general, at a minimum, a 6-point calibration curve that spans the linear instrument detection range is analyzed at the beginning and end of each batch of samples analyzed. In this way, the calibration curve of standards reflects the condition of the instrument while samples are being analyzed.

After the batch has been analyzed the calibration curves are generated. The correlation coefficient should be  $> 0.98$  for the calibration to be accepted. If the correlation coefficient is not  $> 0.98$ , appropriate maintenance and troubleshooting is conducted until the problem is found. Curves with outlier points in the middle ranges suggest that the entire batch was out of control, invalidating this batch and making it necessary to rerun the entire batch from any raw material that remains from the original sample bottle that has been preserved in the lab. Curves with outlier points at the low or high end may exclude these outliers if adequate justification can be made (e.g., concentrations obviously below or above the linear response point of the instrument). Any samples with concentrations of an analyte more than 10% above the top calibration point require appropriate dilution of the primary sample (preserved in the lab) and complete reprocessing and reanalysis of that sample for that particular compound.

#### **B8. Inspection/Acceptance of Supplies and Consumables**

All supplies and consumable materials, such as solvents, reagents, labware, extraction cartridges, and other materials use in this analysis have been subjected to continuous testing as part of this laboratory's research. Only materials that have been found to be reliably free from PFAS contamination will be used. To ensure that these materials remain free of potential contaminants, solvent blanks, matrix blanks, and field blanks will be analyzed with every analytical batch run in this evaluation. Results from these analyses must show that all analytes are  $< \text{LOQ}$  (limit of quantitation). If any of these analyses yield target analyte concentrations that are  $> \text{LOQ}$ , the results of the entire batch will be flagged and a systematic evaluation of the materials used in the entire process will be conducted until the source of the contamination is found. Once the contaminated material has been identified, it will be replaced with a new batch or lot that has been tested to be free from contamination and the entire batch will be rerun.

#### **B9. Non-direct Measurements**

No secondary/existing data will be used for this project so this section is not applicable.

#### **B10. Data Management**

The Data files are the electronic versions of these data. The electronic version of data is calculated by the instrument software and then exported to Excel. The file path(s) for where electronic data is stored will be documented in a laboratory notebook. Raw data (including electronic data on individual PC hard drives and group shared drives) will be backed up to a

network or external drive. All data generated will be maintained Drs. Mark J. Strynar and John Washington until completion of the project. Upon completion, data will be stored in accordance with EPA's record management policy. All instrument data and the Excel spreadsheets will be backed up to network drives routinely and will be archived along with other supporting data and relative correspondence at the completion of the study. Printed data will be referenced in research notebooks, signed and dated in accordance with the Office of Research & Development's Policy and Procedure Manual Section 13.02 on Paper Laboratory Records. Laboratory notebooks will be the record for any procedure conducted in the laboratory and will provide the objective, procedure details, data references and discussion for project development. These entries will give a full and complete statement of the situation being examined, a specific hypothesis relating to the situation, and a brief experimental protocol designed to answer the questions posed in the hypothesis. Data will be recorded from these experiments and a discussion of the results will be presented with conclusions drawn. Any standard, solution, or sample made during these investigations will be marked with a reference number, traceable to a specific entry in the lab notebook. The laboratory notebooks are the property of the EPA and will be stored in accordance with EPA's record management policy.

When shipping samples from NJ DEP to the EPA/RTP or Athens laboratories, NJ DEP will include the CoC sheets for each sample contained within the shipment.

Upon receipt at the laboratory, the sample custodian will check the contents of each shipping container for sample container breakage, and will verify that contents match the shipping lists. After logging in each sample, and signing CoC forms, the samples will be transferred to the proper storage facility.

In the laboratory, the data will be verified and checked at several levels. The instrument operator will generate the calibration curves, apply the curves to detected analytes, and complete a first pass assessment of accuracy of identification of analytes in samples. This will be done onscreen using an automated procedure where the system pages through the data for identification of each analyte in each sample. If adjustments are needed in the integration area, baseline, or peak identification, this will be done manually by the analyst. Electronic output will include a copy of the method, a copy of each calibration curve with equation and fit printed, run list, and a summary of the quantitation for each analyte. As noted above, the electronic files will be stored on the instrument computer and backed up routinely.

The technical reviewer will review data for accuracy in identification and quantification. This reviewer will monitor retention time, mass transition, and qualifier ions.

The LC/MS/MS analytical data for samples are preprocessed and initially reviewed using spreadsheet software; this is a three-step process and is described as follows:

- 1) The LC/MS/MS summary data are electronically transferred into an Excel spreadsheet or exported as a delimited text file that can be read in Excel. This consists of the analysis date, the sample identification number and classification (e.g., blank, QC, calibration, unknown), target

analyte names, analyte and internal standard (IS) peak area counts, analyte/IS peak area ratios, quantitation ion/qualifier ion ratio, and concentration of analyte. The first reviewer assures that this electronic transfer has been made accurately and completely.

2) The first reviewer hand-enters into the spreadsheet any ancillary analytical data required for the Excel spreadsheet to calculate final concentrations, e.g., dilution factors, calibration standard concentration adjustments. Any hand-entered data will be checked by a second analyst. The spreadsheets function as a user friendly interface for data entry and they also can impose uniformity on the reported data by requesting data values in specific units that have a uniform accuracy, i.e., number of significant digits. A straight-forward design of a spreadsheet template also permits all the data from any given analytical run to be reviewed by the task leaders without requiring them to invest time learning new software.

The final analyte concentrations are reported in ng/L for water and pg/g for solid matrices using validated equations that take into account dilution factors, standard purity and salt concentration, and volume of unknown sample used in the analysis.

3) The QA Manager or appropriate designee will review a subset of all laboratory data for accuracy in reporting, transcription, and calculations.

## **C. ASSESSMENTS AND OVERSIGHT**

### **C1. Assessments and Response Actions**

Data audits will be a random check of electronic and hand-entered data conducted by the EPA EMMD QA Manager or appropriate designee. These audits will focus on review of data transcription, calculation, and reporting. The EMMD QAM or designee will report to the researcher any findings requiring corrective action. Any findings requiring corrective action will be noted in the file and discussed in the final report.

Internal quality control measures described in this QAPP, implemented by the technical staff and monitored by the EPA PIs, will give information on data quality on a day-to-day basis. The responsibility for interpreting the results of these checks and resolving any potential problems is shared between the technical staff and the EPA PI. Field and technical staff have the responsibility to identify and report any problems that could affect data quality or the ability to use the data. Any problems that are identified will be reported to the EPA PI, who will document the problem and any loss of samples. Action will be taken by the PIs to control the problem, identify a solution to the problem, and ameliorate losses and correct data, where possible.

Technical systems audits (TSAs) or surveillance audits may be conducted by the EMMD QAM or designee to assess implementation of this QAPP. Any findings will be reported to the respective EPA PIs and corrective actions will be implemented by responsible parties to address those findings.



## C2. Reports to Management

EMMD staff will meet with NJ DEP Project Manager and EPA Region 2's Coordinator periodically to discuss the progress of the project. The EMMD Division Director will be responsible for transmitting data results through EPA Region 2's Coordinator to the NJ DEP. These reports will be provided on a timeline agreed upon by all parties.

Results of any QA audits will be reported as part of the final report. This section will summarize any errors, deficiencies, or deviations from the QA documentation that may or may not have an effect on the data.

## D. DATA VALIDATION AND USABILITY

### D1/D2. Data Review, Verification, and Validation/Verification and Validation Methods

The goal of data verification is to ensure that complete and accurate analytical information is available for all samples analyzed by the laboratory. Data verification begins during the period of analysis and continues through data entry into the Excel spreadsheet. The key personnel of the analytical team will perform the first level of review, ensuring that all data have been validated. The mechanisms used for all data transcriptions and transmissions will be reviewed, and a random subset of all transcriptions checked. For data requiring calculation of results, a random subset of the raw data will be recalculated.

Once the chemical measurement data have been imported into Excel spreadsheet, the following QA/QC checks to validate the data will be carried out:

- Sample ID checks to verify that all Sample IDs with reported data are valid Sample IDs, i.e., they were logged in as received from NJ DEP.
- Missing data checks to verify that all Sample IDs received from the field either had a full set of analytical data reported or were disqualified, as documented in the CoC data.
- Duplicate data checks, to verify that the same analytical data were not imported into the Excel spreadsheet twice for a given sample.
- Out-of-range checks, to verify that all data for data fields limited to a code set did not violate that code set.
- Calculation verification; for any calculations performed within the Excel spreadsheet a random subset of the raw data are calculated using an independent calculation source (Excel spreadsheet) for verification.

Data quality flags will be assigned to each chemical measurement record as needed to identify the quality and usability of the record. Data quality flags will be assigned as documented in each report.

Data verification will also include the evaluation of the results of the QC samples shipped with each batch of unknowns. Since these QC samples will be generated in the lab prior to shipment to the field, these samples will reflect all the sample preparation, shipping, storage, and handling of actual field samples. For laboratory and field blanks, if the background level for a given target analyte is unacceptably high, the target analyte results for all samples affected by this background will be flagged. The recoveries of analytes in these field samples will be an accurate reflection of recovery of analytes throughout the entire process. In addition, the recovery of the IS compounds will demonstrate the method performance on a sample by sample basis. Since the ISs are the labeled version of the “average” analyte of each class, the IS recoveries can be used to effectively account for losses during analytical procedures on a sample by sample basis.

### **D3. Analysis and Reconciliation with User Requirements**

The EPA RTP and Athens PFAS labs will summarize the chemical analyses of the all samples in a final report for NJ DEP that will provide information to determine data quality and the range of target analyte levels found in the samples.

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